INTERNATIONAL STANDARD

ISO 1625

Second edition
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Plastics — Polymer dispersions — Determination of non-volatile matter (residue) at specified temperatures

Plastiques — Dispersions de polymères — Détermination de la matière non volatile (résidu) à des températures spécifiées

Reference number
ISO 1625:1998(E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 1625 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 9, *Thermoplastic materials,* with inputs from ISO/TC 35, *Paints and varnishes,* and ISO/TC 45, *Rubber and rubber products.*

This second edition cancels and replaces the first edition (ISO 1625:1977), which has been technically revised.

In contrast to the previous edition, this new edition is based on the fact that the mass of the so-called “residue” or “dry solids” is not an absolute value, but depends on the conditions used for the determination. To allow the conditions to be adapted to the sample material, four different combinations of temperature and heating time are specified, and the most appropriate combination is selected from these four by agreement between the interested parties.

It is planned to harmonize this standard, if possible, at a future date with ISO 3251:1993, *Paints and varnishes — Determination of non-volatile matter of paints, varnishes and binders for paints and varnishes,* and EN 827:1994, *Adhesives — Determination of conventional solids content and constant mass solids content.*

Annex A forms an integral part of this International Standard.

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Plastics — Polymer dispersions — Determination of non-volatile matter (residue) at specified temperatures

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

1 Scope

This International Standard specifies a method for the determination of the non-volatile matter content of polymer dispersions.

The method is applicable to all polymer dispersions whose residue is chemically stable under the test conditions.

The method is also applicable to formulated polymer dispersions containing fillers, pigments and other auxiliaries.

NOTES

1 This method is suitable for synthetic rubber latices provided heating for a specific period of time is considered appropriate (ISO 124 specifies heating until the loss in mass of a 2 g test portion following successive periods of heating is less than 0,5 mg).

2 The residue from unplasticized polymer dispersions and rubber latices consists essentially of the polymeric material and of small quantities of auxiliaries such as emulsifiers, protective colloids, stabilizers, solvents added as film-forming agents and — especially for rubber latex concentrate — preserving agents. For plasticized samples, the residue by definition normally includes the plasticizer.

3 In-house methods for the determination of non-volatile matter often include drying with infrared or microwave radiation. Standardization of such methods is not possible, since they are not generally applicable. Several polymer compositions tend to decompose during such treatment and therefore give incorrect results.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 123:1985, *Rubber latex — Sampling.*

ISO 124:1997, *Latex, rubber — Determination of total solids content.*

ISO 842:1984, *Raw materials for paints and varnishes — Sampling.*

3 Definition

For the purposes of this International Standard, the following definition applies:

3.1 non-volatile matter: The residue obtained by evaporation (drying) under specified conditions.

4 Principle

A test portion is spread in a thin, even layer in a dish and evaporated in an oven under specified conditions of temperature and time, depending on the material to be tested. The determination is performed at atmospheric pressure.

For certain applications, drying in a vacuum may be preferable. In such cases, the conditions shall be agreed on or the method specified in ISO 124 shall be used.

The non-volatile matter content of the sample is determined by weighing before and after heating.

5 Apparatus

Ordinary laboratory equipment, plus the following:

5.1 Flat-bottomed dish, made of metal, 70 mm ± 10 mm in diameter, height of lip or rim at least 5 mm. Aluminium lids have been found suitable for this purpose.

NOTES

1 For rubber lattices, lipless dishes with covers are recommended.

2 For very viscous polymer dispersions or latices, it is recommended to use aluminium foils about 0,1 mm thick, cut into rectangles of about (70 ± 10) mm × (120 ± 10) mm that can be folded in half, thus allowing the viscous liquid to be spread by gently squeezing the halves together.

5.2 Air oven, capable of maintaining the temperature selected (see annex A) to within ± 2 °C. Unless agreed otherwise (e.g. to use a natural-convection oven), the air shall be mechanically circulated. For round-robin tests, ovens of equivalent construction shall be used by all parties.

WARNING — To protect against explosion or fire, polymer dispersions and latices containing volatile flammable substances should be handled with care. National regulations should be followed.

5.3 Analytical balance, capable of weighing to an accuracy of 0,1 mg.

5.4 Dessicator, containing a suitable desiccant, for example dried silica gel impregnated with cobalt chloride.

6 Sampling

Take a representative sample of the product to be tested, as described in either ISO 123 or ISO 842.

7 Procedure

Carry out the determination in duplicate.

Degrease and clean the dish (5.1). Dry the dish in the oven (5.2) under the selected conditions of temperature and time (see annex A) and allow it to cool to ambient temperature in the desiccator (5.4).

Weigh, to the nearest 1 mg, 1 g ± 0,2 g (m_0) of the sample into the dish and distribute it evenly.

Test portions other than 1 g may be used by agreement between the interested parties. However, the size of the test portions shall not exceed 2,5 g.

Test portions of 0,2 g to 0,4 g, weighed to the nearest 0,1 mg, may also be used. In this case, the drying times given in annex A can be reduced provided it has been established (by measurements on the type of dispersion under test) that the same results are obtained as under the conditions given in annex A.

If a test portion of a size other than 1 g is used, this shall be stated in the test report.

In the case of products that are highly viscous or tend to form skins, distribute the test portion uniformly with a tared wire (for example an uncoated, bent paper-clip) or use aluminium foils (see note 2 to subclause 5.1).

Aqueous systems such as polymer dispersions and latices may splash when heated, due to surface skinning. The thickness of the layer of test material shall therefore be kept as low as possible.

Transfer the dish with the test portion to the air oven preheated to the selected temperature and leave the dish in the oven for the selected length of time (see annex A).

After completing the drying operation, transfer the dish to the desiccator and allow to cool to room temperature. Weigh the dish with the residue to the nearest 1 mg and determine the mass (m_1) of the non-volatile matter (residue).

8 Expression of results

Calculate the non-volatile matter content R, expressed as a percentage by mass of the material under test, using the following equation:

$$R = \frac{m_1}{m_0} \times 100$$

where

m_0 is the mass, in grams, of the test portion;

m_1 is the mass, in grams, of the residue.

Should the two results (duplicates) differ by more than 0,5 % (e.g. 53,7 % and 53,1 %), discard the results and repeat the procedure described in clause 7.

Calculate the mean of two valid results and record the test result to the nearest 0,1 %.

9 Precision

With accurate operation and control, i.e. following the details of the procedure, it is possible to attain the following:

Repeatability

The difference between two test results, each the mean of duplicates, obtained on identical material by the same operator using the same apparatus will not exceed 0,6 % (relative to the mean value of the two test results).

Reproducibility

The difference between two test results, each the mean of duplicates, obtained on identical material by operators in different laboratories will not exceed 1 % (relative to the mean value of the two test results).

10 Test report

The test report shall include the following particulars:

a) a reference to this International Standard;
b) sufficient information to identify completely the sample tested;
c) the conditions used for the determination (temperature and time);
d) the size of the test portion used;
e) the result of the test (clause 8);
f) any deviation from the test method specified;
g) any unusual features observed concerning the behaviour of the sample ;
h) the date and the place of the test.